BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

As rescanning documents *will not* correct images, please do not report the images to the Image Problem Mailbox.

F56

(12) UK Patent Application (19) GB (11) 2 189 127 (13) A

(43) Application published 21 Oct 1987

(21) Application No 8709061

(22) Date of filing 15 Apr 1987

(30) Priority data
(31) 852482 (32) 16 Apr 1986 (33) US

(51) INT CL⁴
A24D 3/06 3/02

(52) Domestic classification (Edition I):
A2C 1E3

(56) Documents cited
GB 1400197 GB 1085582 US 3605759

(58) Field of search
A2C
Selected US specifications from IPC sub-class A24D

(71) Applicant
Hercules Incorporated

(Incorporated in USA—Delaware)

Hercules Plaza, Wilmington, Delaware 19894, United States of America

(72) Inventor
Michael Dennis Leiah

(74) Agent and/or Address for Service
Michael Burnside & Partners,
2 Serjeants Inn, Fleet Street, London EC4Y 1HL

(54) Cigarette filter manufacture

(57) Filter elements for cigarettes are made by treating a substrate such as a polyolefin or cellulose acetate with (A) one or more of sodium bicarbonate, sodium carbonate, potassium permanganate and manganese dioxide, and (B) a solution or dispersion of a non-ionic spin solution which may contain aqueous dispersant such as a polyoxyalkylene derivative of a sorbitan fatty acid ester, a monoester of a polyhydroxyalcohol, or a diester of a polyhydroxy alcohol.

Also exemplified are fiters comprising a substrate, glycerol triacetate and spin solution.

The filters are particularly effective at removal of hydrogen cyanide and nitric oxide from cigarette smoke.

GB 2 189 127 A

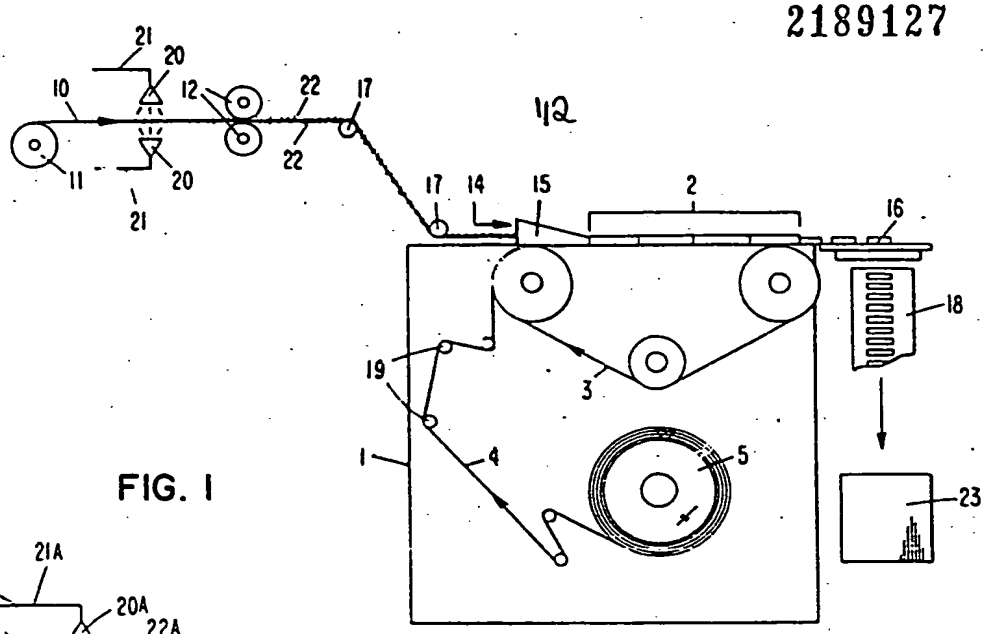

FIG. 1

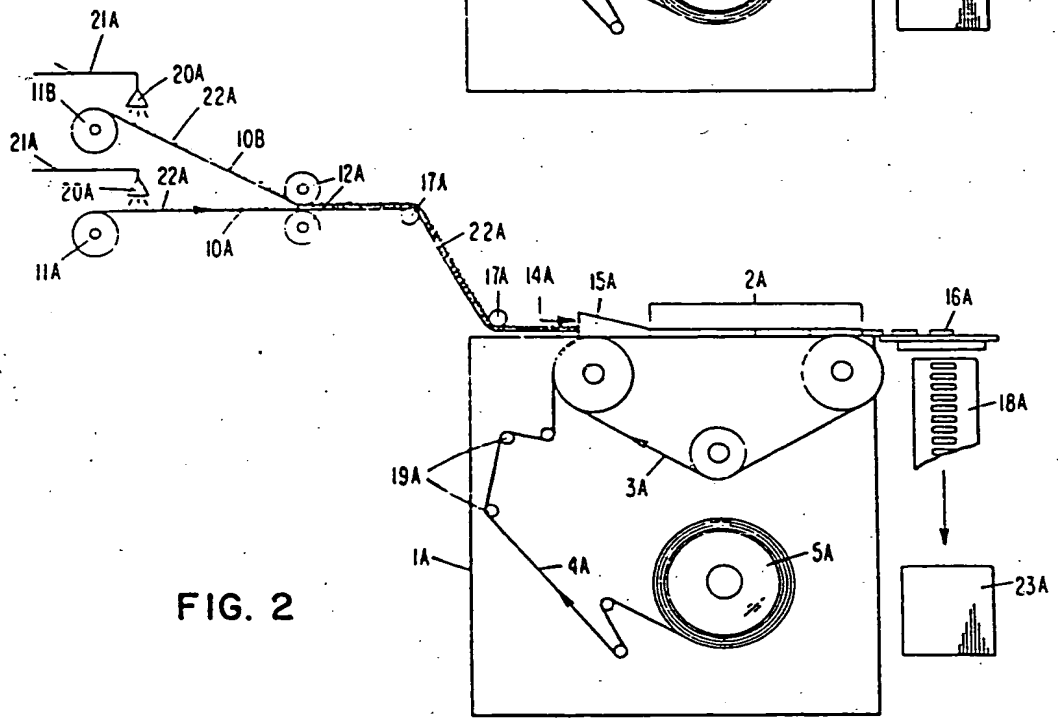

FIG. 2

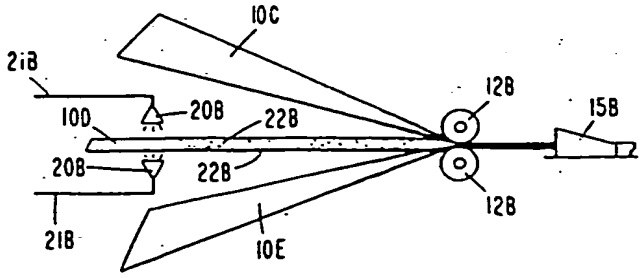

FIG. 3

2189127

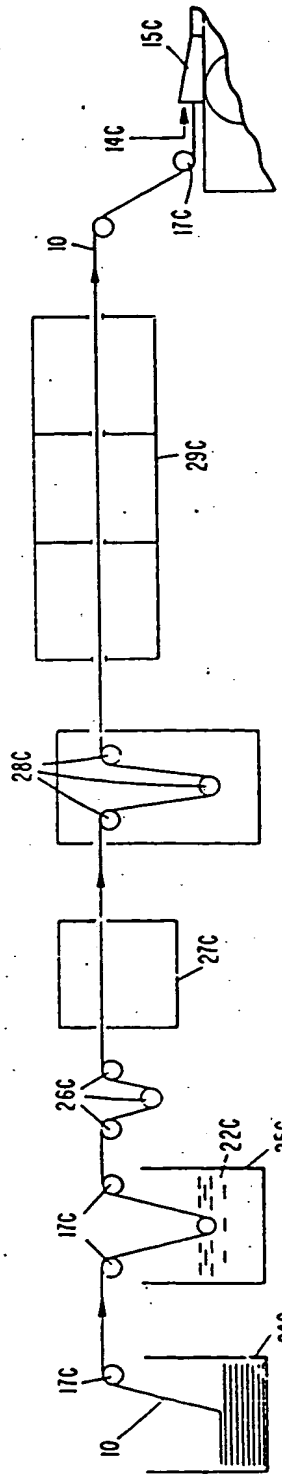

FIG. 4

It is also generally immaterial whether the garniture feed is fabricated, in situ, (i.e. immediately upstream of the garniture) or produced and stored before use and found useful to employ one or more nonwoven fabrics of the same or different fiber composition and denier, particularly when not all of the substrates are used as a carrier or absorbing surface for the defined active modifier component(s).

When nonwoven fabric is used as substrate components for garniture feed, it can usefully comprise up to about 100% and preferably 10%–100% by weight of staple polyolefin (mono-, bi-, or tri-component) fiber identified generally as staple polypropylene, or may consist of webs having filaments of mixed denier, or combination of fibers such as (a) polypropylene/polyethylene, polypropylene/ polyvinylidene chloride, polypropylene/cellulose acetate, polypropylene/rayon, polypropylene/nylon, cellulose acetate/polyethylene, plasticized cellulose acetate, polypropylene/paper; or (b) polypropylene/polystyrene/polyethylene, and the like, in a preferred ratio of about (a) 10%–90%/90%–10% or (b) 10%–90%/45%–5%/45%–5% by weight of fiber.

Suitable fibrillated film as substrate component for use alone or in combination with other substrate components as garniture feed within the present invention are obtained, for instance, in accordance with components commonly disclosed in the art.

For present purposes, a conventional filter rod-making apparatus suitable for present purposes comprises a tow trumpet, garniture, shaping means, wrapping means, and cutting means in accordance with components and processes generally described in the art. If desired, however, modifications can be made to permit in-situ or prior spraying, dipping, printing or other traditional application of one or more modifier components prior to formation of a filter plug, and preferably before drawing through a garniture.

By way of further example, baled sliver or other substrate can be prepared for use by continuous dip coating or by contact with one or more printing roll(s) fed from reservoir(s) of desired active components(s), followed by conventional drying steps effected by nip rolls, heated drying rolls, ovens, and the like, at temperatures generally within the range of about 70°C–125°C.

Generally speaking, nonwoven material from fibers within a wide denier range can be obtained using art-recognized techniques. Preferably such material falls within a weight range of about 10–50 grams per m^2, and a ribbon width of about 4"–12" will generally assure successful passage through the garniture of a conventional filter rod-making apparatus at production speeds.

As above-indicated, the garniture feed may usefully comprise up to about 4 or even more substrate components of identical or different weight, dimensions, bonding, absorption, fiber composition and denier, and can be introduced wholly or partly in register in the garniture. For best results, however, one relatively lightly thermally bonded fabric, tow, sliver or fibrillated film in register with one, or between two nonwoven fabrics is found to offer a high degree of flexibility for adapting the resulting filter element to a variety of market needs, including cost, filter draw, and hardness parameters.

The inclusion of an additional low melting fiber such as polyethylene, combined with other polyolefin fiber as garniture feed is found useful (although not mandatory) for obtaining tow plugs of widely varying bonding and liquid absorption or adsorption properties.

Supplemental components can also be employed, such as solutions, emulsions, suspensions or dispersions of one or more humectants generally exemplified by polyhydric alcohols such as glycerols, glycols, etc.; flavors and perfumes such as ketoses and polysaccharides, including wintergreen, spearmint, peppermint, cinnamon, fruit flavors, etc., and additives as otherwise found in art; medicines, such as menthol, decongestants, etc.

For present purposes, both treated and untreated fabric ribbon can be usefully wrapped using regular plug wrap paper having a weight within a range of about 25–90 g/m^2 or higher, as desired.

Exemplary embodiments of the present invention are further described in the following drawings in which Fig. 1 diagrammatically represents a conventional cigarette filter rod-making apparatus modified to convert substrates prepared in accordance with the instant invention into filter elements, and Figs. 2–4 diagrammatically represent further modifications and improvements within the instant invention, whereby one or more slivers ribbons of nonwovens or other substrates in register or partial register, are easily impregnated with one or more active modifier components by spraying or dipping and the use of multiple substrates favors increased filter element bulk and improved crush resistance, or hardness.

In *Figure 1*, a single continuous substrate such as a fiber tow, sliver, fibrillated film or ribbon of nonwoven fabric (10) is fed from feed reel (11) or a bale (not shown) and through spray heads (20) feedably connected to feed lines (21) from outside sources (not shown) to apply separate active modifier components (22). The treated substrate (10) is then dried by air drying means (not shown) and by passing over drying rolls (12), to a desired degree of dryness, and led by guide rolls (17) into a garniture trumpet (15) and garniture (14) of a cigarette filter rod manufacturing apparatus (1) comprised of a garniture section (2) including (but not showing)

SPECIFICATION

Method and device for controlling hydrogen cyanide and nitric oxide concentration in cigarette smoke

This invention relates to a method for making a chemically-treated filter element for selective filtration of cigarette smoke.

Fiber based filter elements are well-known and have been used in the cigarette manufacturing field for at least 40 years. While various synthetic fiber and fiber mixtures have been tried and evaluated in the market place, most cigarette filter elements continue to include cellulose acetate-based fibers.

Synthetic fibers, particularly fibers of polyolefins such as polypropylene are easily drawn to a much smaller denier than cellulose acetate-based fibers, offering improved filter efficiency without simultaneous loss of strength needed for crimping and high speed production. Such fibers, however, also have disadvantages. These stem mainly from the fact that a substrate, such as a open or bloomed tow is relatively inert and not readily wetted or softened by most adhesive/plasticizer or other hydrophilic-type modifier formulations.

The incorporation of chemically-active ingredients in order to achieve selective filtration of cigarette smoke has led to various difficulties connected with the substrate characteristics, uneven distribution, as well as interrelationships between filter element efficiency, and the dimensional stability, resiliency and hardness of the resulting filter element. Polyolefin fiber-containing substrates (i.e. tows and slivers) fed into a filter rod-making apparatus have demonstrated a significant negative correlation between pressure drop (resistance to draw) and dimensional stability or hardness of the filter element. In addition, it is often very difficult to avoid jamming of impregnated polyclefin or other synthetic substrates fed at high speed into a conventional filter rod-making apparatus, particularly substantial amounts of modifier components such as adhesives, humectants, flavors, medicines, absorbents, adsorbents, and the like, into or onto the garniture feed. This is due, in part, to an inherent deficiency in lubricating properties of many additive compositions used in the cigarette filter art.

For instance, U.S. Patent 3,428,056 points out that the addition of water-soluble inorganic salts that effectively remove hydrogen cyanide from cigarette smoke lead to non-uniform powder distribution and poor filtration efficiency. That patent attempts to deal with the problem by using polyolefin powders that have been coated with inorganic salts such as sodium and potassium carbonates, the powders being vibrated onto cellulose acetate tow that has been previously treated with a bonding agent such as glycerol triacetate and that is the n compacted and wrapped to form a filter rod.

According to the invention, a method for making a chemically-treated filter element for cigarettes in which a cigarette filter element obtained from a conventional filter-rod-making apparatus using, as garniture feed, a polyolefin or cellulose acetate-containing substrate, or a combination of the two, is treated with an active modifier component such as sodium carbonate and with a solution of glycerol triacetate in water or alcohol, characterized in that the modifier components comprise in one or more of sodium bicarbonate, sodium carbonate, potassium permanganate and manganese dioxide, a non-ionic solution or dispersion of the non-ionic solution or dispersion that also contains as an aqueous dispersant a polyoxyalkylene derivative of a sorbitan fatty acid ester, a mono-ester of a polyhydroxyalcohol, or a diester of a polyhydroxy alcohol, or a mixture of the said dispersants, and the non-ionic solution or dispersion is applied to all or part of the substrate in the garniture feed.

The non-ionic solution or dispersion will be referred to as a non-ionic "spin solution" in acordance with usage in the art.

Preferably the amount of the modifier components is applied is about 2%–10% of the dry filter weight with the amount of the glycerol triacetate comprising about 1%–10% of the dry filter weight.

Preferably, the surfactants in the non-ionic spin solution are ethoxylates, carboxylic acid esters, glycerol esters, polyoxyethylene esters, anhydrosorbitol esters, ethoxylated anhydrosorbitol esters, ethoxylated natural fats, oils and waxes, glycol esters of fatty acids, polyoxyethylene fatty acid amides, polyalkylene oxide block copolymers, and poly(oxyethylene-co-oxypropylene).

The substrate the method according to the invention may be a fiber-containing or fibrillated film-containing component conventionally used as garniture feed in a filter rod-making apparatus, including (a) a fiber tow, (b) a sliver, (c) a ribbon of a nonwoven material or (d) a web of fibrillated film, which may be introduced alone or in complete or partial register for insertion into the garniture.

Garniture feed within the instant invention can conveniently include up to four substrate components, with desired active components, preferably individually applied onto one or both faces of selected substrates, the manner and number of faces treated depending upon the desired filter efficiency, taste, feel, hardness, and draw of the filter.

means for shaping and retaining the substrate feed, wrapping means, and cutting means for converting the wrapped plug or rod into filter element (16); the wrapping means is conveniently supplied with tow wrap from wrap feed reel (5) supported by support rolls (19) and moved onto a continuous garniture belt (3) for introduction into the apparatus.

The apparatus, as described, comprises conventional means for sealing the tow wrap around a filter plug (not shown) which is cut by cutting means into cylindrical filter elements (16) of desired length (normally 90 mm), before removal by filter chute (18) (shown in fragment) for packing in container (23).

Figure 2 diagrammatically demonstrates a further arrangement for separately applying active modifier component(s) onto a garniture feed or substrate whereby spray heads (20A) fed by connecting feed lines (21A) separately apply active modifier components (22A) (identical or otherwise) onto different substrates (10A, 10B), fed in register and dried using air and heated rolls (12A) before passage through garniture (14A) of rod-making apparatus (1A), to form filter elements (16A) as before. Substrates (10A and 10B), are fed from feed rolls (11A) and (11B) or bales (not shown) and conveniently brought into register at heated nip rolls (12A), then guided by guide rolls (17A) into garniture (14A), the garniture feed or substrate components shown being similarly defined by arabic numbers in each of Figs. 1–3.

Figure 3 diagrammatically demonstrates a further modification of the equipment and process of Figs. 1 and 2, whereby several substrates of the same or different types (10C, 10D, and 10E) from reels or boxes (not shown) are fed through a nip created by heated rolls (12B), the middle substrate (10D) being of different width and preferably having higher absorption or adsorption properties for retaining active components (22B), then the two external untreated substrates (10C and 10E). As shown, substrate (10D) is sprayed on both sides to selectively expose it to one or more active modifier components (22B) applied by spray heads (20B) fed from feedlines (21B), one substrate (10E) preferably being wider and arranged so as to catch surplus drip or misdirected active components not retained or captured by ribbon (10D), all three substrates are then air dried by passing in register through heated nip rolls (12B), as before, and directed by guide rolls (not shown) into the garniture of a filter rod apparatus in the manner of Figs. 1 and 2.

Figure 4 is a diagrammatic representation of a further modification in which one or more substrates (not shown) can be separately fed from a bale or box (24C), passed over guide rolls (17C), and dipped into a reservoir (25C) containing active modifier component (22C), then passed through nip rolls (26C), through a heating oven (27C), through drawer rolls (28C) and a three step drying oven (29C), to garniture (14C) of a cigarette rod manufacturing apparatus in the manner of Figs. 1–3, supra or boxed for future use.

Where a continuous fiber tow is used as a substrate component, preparation of the tow is conveniently carried out in the usual way by drawing the fiber from one or more creels through a fluid bulking or texturing jet and then handled as noted above.

Substrates which are employed in the above manner can usefully be of a variety of synthetic filaments as noted above. Thus, it is possible to use polyesters, polyamides, acrylics, as well as polypropylene or cellulose acetate materials. Due to its relatively low density, compared to other synthetic fiber-forming material and excellent spin properties, combinations of filament-forming copolymers of propylene with ethylene or other lower olefins monomers are preferred tow, ribbon and fibrillated film material.

The bulk denier of a tow for carrying out the present invention can conveniently fall between about 2,000 and 10,000. As noted above, this substrate can be supplied as a crimped fiber from a single creel or bale, or a composite of several creels or bales combined and passed through a fluid jet simultaneously. For best performance as cigarette filters, however, it is preferred that at least some of the tow be substantially untwisted and untexturized prior to entering a fluid jet.

The invention is further illustrated by the following Examples.

Example 1

(A). Isotactic polypropylene staple fiber (4.5 dpf and 1.5" cut) having a "Y" cross section and a flow rate of 40±5g/10 min., is carded into a web weighing about 0.18 grams per yd.^{2}. The web is transferred onto a continuous fiberglass belt and lightly thermally bonded using a hot diamond-patterned calender at 140°C./40 psi roll pressure to obtain a nonwoven fabric which is die cut into 12 inch width test ribbon substrate hereafter identified as TS-1.

(B). Spun drawn 2.5 denier cellulose acetate yarn (circular cross section) obtained under conventional commercial spin conditions is unwound in parallel from a roll off creel under 0.01g/denier tension and combined to form a fiber tow. The tow is then fed through a bulking jet using steam at 70 psi (107–110°C), the resulting spread tow substrate being hereafter identified as TS-2.

Compositions within the general definition of active modifier components A1–A6, and active modifier components B1–B2, supra, are applied as finish compositions to substrates TS-1 and

TS-2 in the manner indicated in the Examples, the finish compositions used consisting of the following:

Finish		*Compositions*
A1	5 wt %	Sodium carbonate
	5 wt %	Atmos™ 300[1]
	5 wt %	Monolaurate ester of polyoxyethylated sorbitol[2]
	85 wt %	Water
A2	5 wt %	Sodium acetate
	5 wt %	Atmos 300
	5 wt %	Tween 20
	85 wt %	Water
A3	5 wt %	Sodium bicarbonate
	5 wt %	Atmos 300
	5 wt %	Tween 20
	85 wt %	Water
A4	5 wt %	Potassium permanganate
	5 wt %	Atmos 300
	5 wt %	Tween 20
	85 wt %	Water
A5	5 wt %	Sodium bicarbonate
	2.5 wt %	Atmos 300
	2.5 wt %	Tween 20
	90 wt %	Water
A6	5 wt %	Sodium bicarbonate
	0.25 wt %	Atmos 300
	0.25 wt %	Tween 20
	94.5 wt %	Water
B1	1 wt %	Glycerol triacetate
	5 wt %	Atmos 300
	5 wt %	Tween 20
	89 wt %	Water
B2	5 wt %	Glycerol triacetate
	5 wt %	Atmos 300
	5 wt %	Tween 20
	85 wt %	Water

*1. Nonionic surfactant commercially obtained under this mark from ICI Americas.
*2. Commercially obtainable under the mark Tween 20™ from ICI Americas.

Example II

A. A twelve inch (12") wide ribbon of continuous non-woven polypropylene material (TS-1) from Example I is roll dipped into a bath of A1 finish in the general manner shown in Fig. 4, passed through heated nip rolls, oven dried, and hand rolled to form a filter rod of standard width which is cut into 27mm filter elements (0.18 g and 24.35mm circumference) identified as F-1 for testing purposes.

B. A twelve inch (12") wide ribbon continuous nonwoven material (TS-1) is hand rolled as in Example I, but without the dipping or drying steps of Example II A. The resulting filter rod is then cut into 27mm (0.18 g) lengths, as before, and hereafter identified as FC-1 for control testing.

C. A filter testing device[3] comprising a valved constant pressure HCN/N_2 gas source is flowably secured on the down stream side by glass and Tygon tubing to a plastic filter holder which is connected, on its downsteam side, to a Drager Tube[4] packed with $HgCl_2$ and methyl red indicator for HCN colormetric determinations.

D. Filter elements obtained from the F-1 and FC-1 non-woven ribbons are tested for HCN removal by mounting representative filter elements and passing an HCN/nitrogen gas mixture (100 ppm HCN) through the test filter elements at the rate of 50 ml/minute for a period of twelve (12) minutes per test. Test results are reported in Table I infra.

*3. Set up under hood.
*4. Obtained from National Drager inc., Pittsburg, Pennsylvania.

Example III

Example II A–D. are repeated using the A2 and A3 finishes as active modifier components with corresponding controls. The samples, identified as F-2, F-3, FC-2 and FC-3 are tested using a Drager Tube as described in Example II and the results reported in Table I.

Example IV

Test filter elements are prepared using a twelve inch (12") crimped tow web of plasticized cellulose acetate, of 2.5 dpf circular cross section, dipped into A4 finish, dried, hand rolled in the manner of Examples II and III and a 27mm cut filter element tested for HCN removal, using the same Drager Tube as before. Test results are reported in Table I as G-1 and GC-1.

Example V

Camel light tobacco rods are paper wrapped to 27mm test filter elements obtained by dipping (30 seconds) twelve inch (12") crimped fiber tows comprised of 4.5 dpf polypropylene fiber ("y" cross section) into A5 and A6 finishes respectively, then air dried and passed through a standard filter rod making apparatus. Five test filter cigarette of each are smoked on a Borgwaldt smoking machine[*5] and the main-stream gasses analyzed for HCN, using the Drager tube as before. The average results are reported in Table I as C-1 and C-2 with controls CC-1 and CC-2 respectively.

*5. Eight two-second puffs (35 ml).

Table I

Sample	Flow Rate ml/minute	Modifier Component	Time*7	%HCN Removed
F-1	50	A1	12	100
FC-1*6	50	–	12	0
F-2	50	A2	12	96
FC-2*6	50	–	12	0
F-3	50	A3	12	100
FC-3*6	50	–	12	0
G-1	50	A4	12	100
GC-1*6	50	–	12	0
C-1	35	A5	8	61
CC-1*6	35	–	8	0
C-2	35	A6	8	69
CC-2*6	35	–	8	0

*6. Controls

*7. In minutes

Example VI

Two twelve inch (12") of TS-1 nonwoven ribbons, as described in Example II B, are dipped into B1 and B2 finish compositions (ref. pg. 11–12) respectively, dried, hand rolled and cut to obtain 27mm length filters as before. The test filters are individually tested, using the system described in Example II except that the Drager Tube is prepacked with a Cr(V1) catalyst and P,P¹-diamino, m,m¹-dimethoxy biphenyl as a color indicator for detecting residues of nitrous oxide (NO). The test gas contains 100 ppm NO in nitrogen gas, with exposure for 30 minutes at a 50 ml/minute flow rate. Test results for S-1 and S-2 including controls S-1C and S-2C are reported in Table II.

Table II

Sample	Flow Rate ml/minute	Modifier	Time	%NO Removed
S-1	50	B1	30	27
S-1C	50	–	30	0
S-2	50	B2	30	39
S-2C	50	–	30	0

Example VII

Two bulked polypropylene fiber tow substrates (TS-2) obtained from Example 1B are separately sprayed to saturation with A2 and B2 finish compositions using the modified apparatus described schematically in Fig. 2, then air- and roller-dried, and the tow introduced into the garniture of a filter rod-making machine as described in Example II. Randomly chosen 27mm filter elements obtained thereby (average weight of .18 g) are then tested for NO and HCN removal. The test results are found comparable to those reported in Table 1 using A2 and in Table II using B2 modifiers.

CLAIMS

1. A method for selectively removing or controlling concentrations of toxic gas components within cigarette smoke by utilizing as a filtor element, the product of a filter rod-making apparatus, using as garniture feed, at least one polyolefin-containing substrate characterized in that said

substrate is treated with an effective amount of at least one active modifier component comprising at least one member selected from sodium bicarbonate, sodium carbonate, potassium permanganate, manganese dioxide, dissolved or dispersed in a nonionic spin solution.

2. The method of claim 1 wherein the the nonionic solution comprises a polyoxyalkylene derivative of a sorbitan fatty acid ester.

3. The method of claim 1 wherein the nonionic solution comprises a fatty acid monoester of a polyhydroxy alcohol.

4. The method of claim 1 wherein the nonionic solution comprises a fatty acid diester of a polyhydroxy alcohol.

5. The method of claim 1 wherein the nonionic solution comprises a fatty acid diester of a polyhydroxy alcohol.

6. The method of claim 2 wherein the (A) and (B) modifier components are applied to separate substrate surfaces, and said substrate comprises at least one of (a) an open fiber tow, (b) a sliver, (c) a ribbon of nonwoven material or (d) a web of fibrillated film.

6. The method of claim 1, wherein (A) and (B) modifier components are applied to opposite sides of a polyolefin containing substrate.

7. The method of claim 1, wherein the (A) and (B) modifier components are separately applied to different substrates by dipping or spraying.

8. The method of claim 1 wherein polyolefin-containing substrate is pretreated by corona or plasma discharge before application of active modifier component.

9. The method of claim 1, wherein the garniture feed is a nonwoven material in combination with at least one other substrate.

10. A filter element obtained in accordance with the method of claim 1.

11. A cigarette comprising a tobacco rod in serial combination with a filter obtained in accordance with claim 10.

12. A method of removing or controlling toxic gas components in tobacco or cigarette smoke, substantially as hereinbefore described with reference to the accompanying drawings.

13. A method of removing or controlling toxic gas components in tobacco or cigarette smoke, substantially as described in any of the foregoing Examples.

14. A filter element manufactured substantially as hereinbefore described with reference to the accompanying drawings.

15. A filter element manufactured substantially as described with reference to the foregoing Examples.

16. A method of making a filter element substantially as hereinbefore described with reference to the accompanying drawings.

17. A method of making a filter element substantially as described in any of the foregoing Examples.

Printed for Her Majesty's Stationery Office by Burgess & Son (Abingdon) Ltd. Dd 8991685, 1987.
Published at The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.